

The influence of the moisture content on the electrical resistance of two types of cork stoppers

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Abstract

The relationship between the log of the electrical resistance (ER; measured using pin electrodes) and the moisture content (MC) have not been reported in any form of cork. That is important for the cork stoppers industry because it should help in the design and verification of more precise devices for measuring cork moisture content. In this study, using linear regression techniques, different regression models of the type $\log(\text{Log}(R) + 1) = axh + b$ were derived to describe the relationship ER-MC, that was measured using pin electrodes on two types of cork stoppers [natural (N) and agglomerate (AG)].

The results obtained show that in the models proposed, the moisture content of AG cork stoppers can be estimated with an error of $\pm 0.3\%$, while that of N stoppers can be estimated with an error of 0.5% . Neither the geographical origin of the N corks nor the surface treatment to which the AG corks were subjected significantly affected the proposed models.

Therefore, the moisture content of cork stoppers could be measured at the industrial scale using electrical resistance-type moisture meters.

Key words: cork, electrical resistance, moisture content.

Resumen

Influencia del contenido de humedad sobre la resistencia eléctrica de dos tipos de tapones de corcho

La relación entre el logaritmo de la resistencia eléctrica (ER) y el contenido de humedad (MC) no ha sido aún reportada para el corcho. Su estudio es importante para la industria de los tapones porque mejora el diseño y la verificación de los dispositivos que miden su contenido de humedad. En el presente estudio, se determinan por regresión lineal diversas ecuaciones del tipo $\text{Log}10(\text{Log}10(R) + 1) = a.h + b$ que describen la relación ER-MC, y que fue medida con electrodos de tipo aguja («*pin electrodes*») en dos tipos de tapones, naturales (N) y aglomerados (AG).

Los resultados obtenidos muestran que el uso de los modelos propuestos predice el valor de humedad de los tapones de corcho aglomerado (AG) con un error máximo de $\pm 0,3\%$ y de los tapones de corcho natural (N) con un $\pm 0,5\%$ (N). Ni la procedencia de los tapones de corcho natural (N) ni el tratamiento superficial en los tapones de corcho aglomerado (AG) tienen incidencia significativa en los modelos planteados.

Nuestros resultados concluyen que el contenido de humedad de los tapones de corcho puede ser medido a escala industrial usando medidores de humedad por resistencia eléctrica.

Palabras clave: corcho, resistencia eléctrica, contenido de humedad.

Introduction

The anatomical structure and chemical composition of cork determine its thermal, acoustic, resilience and

water-impermeable properties. Its hydrophobic nature, stability in the presence of hydroalcoholic solutions, resilience and high friction coefficient are the reasons why cork has been used since ancient times to make stoppers. Even today, this is the most noble and profitable material in use. Owing to its low thermal conductivity and its behaviour sound-absorbing ability,

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cork has also been widely used to cover floors, walls and ceilings in houses, auditoriums, and even vehicles.

Cork is a hygroscopic material. Its behaviour under changing hygrothermal conditions has been described by González Adrados (1994) and Gil and Cortiço (1998). These authors reported that the moisture content of raw cork at 20°C fluctuates between 6 and 10%, depending on the relative humidity (RH) of the air. For stoppers, the range of variation is smaller, from 5 to 9% for those made of natural cork and from 4 to 8% for those made of agglomerate cork. Knowing the moisture content of cork is important since it influences its mechanical behaviour and the microbiological status of stoppers.

The electrical insulating properties of cork depend largely on its moisture content. The majority of studies on electrical properties of cork (Lança *et al.*, 2006, 2007; Marat-Mendes and Neagu, 2003, 2004) have been orientated towards the characterisation of its electrical and dielectric properties with a view to its use as electrical insulator in industry. However, the electrical resistance-moisture content (ER-MC) curves have not been reported in any form of cork. Resistance hygrometers have been used to determine the moisture content of non-woody materials. For example, Muehlbacher and Taylor (2009) used this technique with yellow-poplar bark. However, they did not determine the ER-MC for this material. They proposed correction factors for the displayed values in hand-held moisture meters calibrated using the ER-MC curve for Douglas fir.

With respect to the curve fitting process for determining the ER-MC curves of wood, the literature contains many expressions. The regression model used in the present study was proposed by Samuelsson (1990): $\log(\log(R) + 1) = axh + b$, where R is the electrical resistance ($M\Omega$) and h is the moisture content (%). This expression was used in an extensive work performed by Forsén and Tarvainen (2000), who determined the coefficients a and b for different types of European

wood (Scots pine, maritime pine, spruce, European oak, birch, beech, alder and larch) and demonstrated its viability in modelling the variation of electrical resistance associated with the moisture content.

Now, there is much market interest in obtaining these curves for cork, because of the requirement to install quality control systems based on ISO 9000 in the cork stopper industry. This system requires the introduction of protocols for verify the quality of measurements made by hand-held electrical resistance-type moisture meters.

The aim of the present work was to obtain the ER-MC curves for different types and quality of cork stoppers. Curves should help in the design and verification of more precise devices for measuring cork moisture content.

Material and methods

Stoppers

The experimental material included four types of commonly marketed stopper (Table 1), two made of a single piece of natural cork (N in Table 1), and two agglomerate corks (AG in Table 1).

In order to determine the influence of the origin of the raw material on the behaviour of the stoppers, N cork samples of the highest commercial quality were chosen from Andalusia (A) and Catalonia (C). These samples were selected such that their density was the most homogeneous possible.

The AG cork stoppers examined were manufactured by the fragmentation of cork into small particles ($0.25 \text{ mm} < d < 8 \text{ mm}$) and their later agglomeration using a polyurethane adhesive and extrusion. Two types of AG stoppers were studied. One type had undergone surface treatment with silicon and paraffin (T) the other type had not (NT).

Table 1. Characteristics of the material examined

Type of stopper	Abbreviation	Diameter (mm)	Length (mm)	Density (kg/m^3)
Natural cork in one piece; quality «A»; origin Andalusia; no surface treatment	N/A	24	44	153.85 ± 5.44
Natural cork in one piece; quality «A»; origin Catalonia; no surface treatment	N/C	24	44	154.82 ± 5.06
Agglomerated cork; surface treated with paraffin and silicone	AG/T	23	44	290.68 ± 0.34
Agglomerated cork; no surface treatment	AG/NT	23	44	292.72 ± 0.61

All the studied stoppers had the most common market dimensions: length 44 mm, and diameter 24 mm for the N stoppers, and 23 mm for the AG stoppers (Table 1). Fifteen stoppers of each type were studied (after measuring, three of them were discarded due to anomalous measurements based on internal defects not detected externally).

Conditioning

After weighing, three stoppers of each type were placed in four climate chambers providing the following ambient conditions: 20°C/RH 40 ± 5%, 20°C/RH 65 ± 5%, 20°C/RH 80 ± 5% and 25°C/RH 85 ± 5%. These chambers provided the conditions required to cover the most common moisture content range of cork stoppers in normal use.

The chambers were monitored weekly, recording the gain or loss of mass experienced by each stopper. Equilibrium was deemed to have been reached when the weekly mass variation was ≤ 0.1%. The stoppers were kept in the chambers until all their masses had stabilised (four months).

The stopper masses were determined using a METTLER TOLEDO (Delta Range PB 303) balance with a resolution of 1 mg. Both the chambers and the balance were subjected to the periodic calibration and maintenance contemplated in the ISO 17025 Manual of Laboratory Quality.

To prevent any loss or gain in moisture content during the measurement process, all the stoppers were placed in small, sealed plastic transport containers previously conditioned in the same climate chambers. After their introduction into their container, the stoppers from the 25°C/HR ± 5% chamber were allowed to become thermally conditioned at 20°C over 2 h in the 20°C/HR 80% ± 5% chamber.

Measurement of electrical resistance

The electrical resistance of each stopper was measured using an AGILENT 4339B high resistance meter (range 10³-10¹⁵ Ω, accuracy 0.5%, display resolution 5 digits). The measuring specifications were:

- Measuring voltage: 10 v.
- Measuring temperature (material and laboratory): 20°C.
- Measuring delay: 5 s.

The electrodes were non-insulated steel needles:

- Diameter: 1.8 mm.
- Length: 25 mm.
- Separation between electrodes: 8.5 mm.

The 8.5 mm separation between the electrodes was chosen since it is commonly employed in the cork industry given that the small diameter of stoppers (as small as 23 mm) prevents the use of that habitually employed in the wood industry (separation between electrodes 30 mm).

To take readings, the electrodes were introduced into the stoppers in the following manner:

- For measurements perpendicular (transversal) to the grain (RT): introduced 20 mm into the base, perpendicular to the growth rings (N type stoppers), avoiding visible pores and anomalies.

- For measurements parallel to the grain (RL): introduced 11 mm on the lateral surface, parallel to the pores (N type stoppers), avoiding visible pores and anomalies.

All measurements were taken after verifying the high resistance meter using a TINSLEY 4721 decade box (in possession of an ISO 17025 calibration certificate).

The moisture content of each stopper was taken following the measurement of its electrical resistance by drying it in an oven at 103 ± 2°C until a constant mass was reached.

Curve fitting was carried out using Statgraphics Centurion XV software, which provided the model coefficients by linear regression techniques. Three anomalous measurements (one for each of the N/A, N/C and AG/T groups) were discarded from analysis.

Results and discussion

Table 2 shows the results for coefficients «*a*» and «*b*», calculated by means of linear regression fitting using the regression model proposed by Samuelsson (1990):

$$\log(\log(R)+1) = axh+b$$

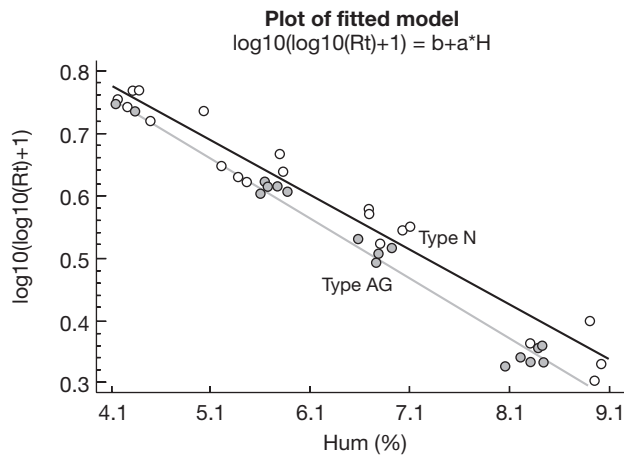
where *h* is the moisture content expressed as a percentage, and *R* the electrical resistance expressed in MΩ. Table 2 also includes the value of the coefficient of determination (*R*²) of each model.

All the models and their coefficients, included in Table 2, were highly statistically significant (*p* < 0.0000).

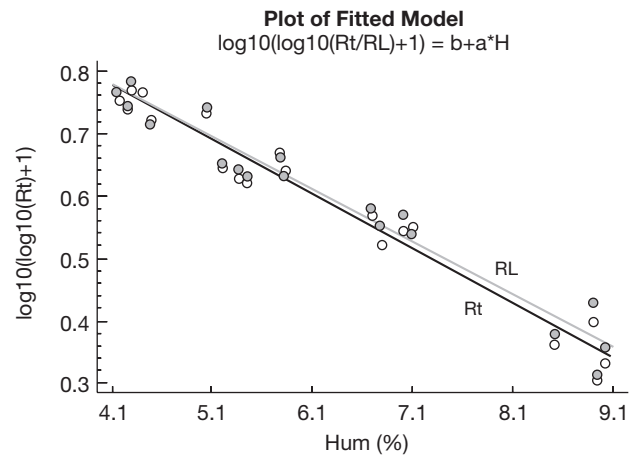
Figures 1-3 reflect the results of Table 2 in graphical format.

Table 2. Regression results

Type	No. samples	Perpendicular to the grain (RT)			Parallel to the grain (RL)		
		a	b	R ²	a	b	R ²
N	22	-0.0872 ± 0.0045	1.1332 ± 0.0287	0.95	-0.0845 ± 0.0044	1.1238 ± 0.0281	0.95
N/A	11	-0.0893 ± 0.0068	1.1524 ± 0.0461	0.95	-0.0863 ± 0.0070	1.1374 ± 0.0474	0.94
N/C	11	-0.0844 ± 0.0059	1.1128 ± 0.0357	0.96	-0.0806 ± 0.0054	1.1017 ± 0.0327	0.96
AG	23	-0.0954 ± 0.0025	1.1464 ± 0.0161	0.99	-0.0866 ± 0.0020	1.1145 ± 0.0131	0.99
AG/NT	12	-0.0939 ± 0.0032	1.1413 ± 0.0208	0.99	-0.0868 ± 0.0031	1.1162 ± 0.0199	0.99
AG/T	11	-0.0969 ± 0.0039	1.1509 ± 0.0252	0.98	-0.0863 ± 0.0029	1.1127 ± 0.0192	0.99

**Figure 1.** Regression results (RT, N and AG stoppers).

The results show that, for the same type of measuring direction (RT or RL), significant differences exist between the models calculated for N and AG stoppers (Table 2, Fig. 1). However, within each general stopper type (N or AG), and for the same measuring direction (RT or RL), neither the surface treatment applied (T or NT) nor the place of origin (A or C) had any signi-

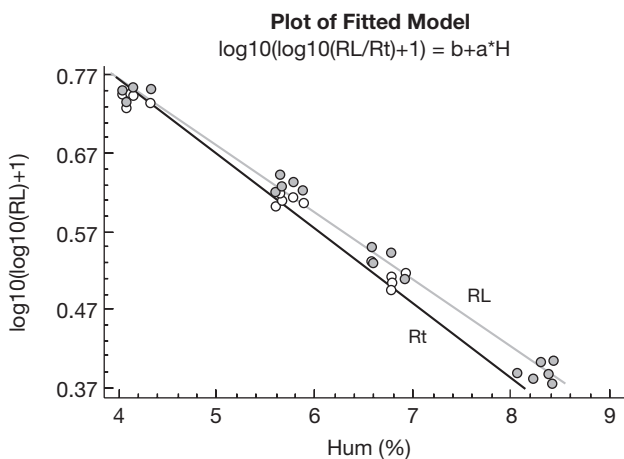
**Figure 2.** Regression results (RT vs. RL for N stoppers).

ficant effect on the calculated coefficients or coefficient of determination (Table 2).

The influence of the measuring direction (RT or RL) only appears to be significant when dealing with AG stoppers (Table 2, Fig. 2 and 3).

Since all measurements were taken at 20°C, the effect of the temperature of the cork on the electrical resistance—and thus on the moisture content—remains unknown.

Figure 4 shows the distribution of errors (transversal measurement, RT) for the estimated values. The estimation of the moisture content of cork stoppers by this electrical method thus has a maximum error of $\pm 0.3\%$ for AG stoppers, and of $\pm 0.5\%$ for N stoppers. Both are very adequate for industrial purposes, being much better than those normally used in wood measurements (between ± 1.0 to $\pm 1.5\%$).

**Figure 3.** Regression results (RT vs. RL for AG stoppers).

Conclusions

The present results show that, using the proposed models (see Table 2), the moisture content of natural (N)

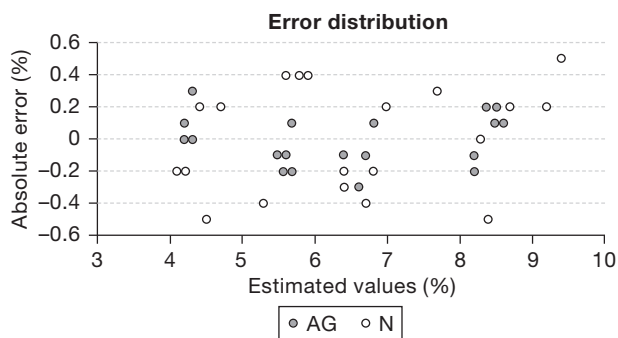


Figure 4. Distribution of absolute error of estimation (RT direction).

and agglomerated (AG) cork stoppers can be estimated with a very small error ($\pm 0.5\%$ and $\pm 0.3\%$ respectively).

Neither the origin of the cork of the natural (N) stoppers nor the surface treatment of the agglomerated (AG) stoppers appears to have any significant influence on the proposed models in Table 2.

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